

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Applicants: Yasushi TOHI et al.
Serial No: 10/550,021 Group: 1713
Filed: September 23, 2005 Examiner: Caixia Lu
For: PROCESS FOR PRODUCING OLEFIN POLYMERS

DECLARATION UNDER 37 CFR 1.132

Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Kenji Sugimura, declare and state that:

1. In March 1987, I was graduated from Osaka University, Faculty of Engineering, Department of Applied Chemistry and received a degree of Bachelor of Engineering from the same University. In March 1989, I was graduated from the master course of the same University, Institute of Engineering, majoring in Applied Chemistry, and received a degree of Master of Engineering from the same University. Since 1989, I have been an employee of Mitsui Petrochemical Industries, Ltd. (now Mitsui Chemicals, Inc.) and till the present time I have been engaged in Research of olefin polymerization catalyst.

2. I am a co-inventor of the invention described in the specification of the above-identified application.
3. I have carried out the following Experiments in order to demonstrate the superior and unexpected results of the present invention.

Experiment B

[Preparation of a catalyst solution]

In a glass flask flushed fully with nitrogen, 0.001 millimole of diphenylmethylenecyclopentadienyl (2,7-di-tert-butylfluorenyl)zirconiumchloride was charged, and a catalyst solution was prepared by adding a toluene solution containing MAO made by Albemarle Corporation in the amount of 0.25 millimole equivalent of Al to the flask.

[Polymerization]

In a stainless-steel autoclave of 2-liter volume fully flushed with nitrogen, 800 milliliter of hexane and 200 milliliter of 1-octene were charged into the autoclave. After increasing the temperature of autoclave content to 145°C, the total pressure was set at 3 MPa-G by charging 500 milliliter of hydrogen and feeding ethylene. Next, 0.3 millimole of

triisobutylaluminum and the catalyst solution prepared as above were pressed into the autoclave by nitrogen pressure, and polymerization was started by setting the rotation of stirrer at 400rpm. Thereafter, polymerization was performed at 150°C for 30 minutes by keeping the total pressure at 3 MPa-G by continuously feeding ethylene only. After stopping the polymerization by adding a small amount of ethanol to the reaction system, unreacted ethylene was purged. By pouring the obtained polymer solution into a large excess of methanol, a polymer was precipitated. After recovering the polymer by filtration, it was dried at 135°C overnight under reduced pressure.

As a result, an ethylene-1-octene copolymer was obtained with a yield of 40.6 g. The obtained polymer had a density of 902 (kg/m³), MFR₂ = 3.25 (g/10 min), MFR₁₀ = 23.1 (g/10 min), MFR₁₀/MFR₂ = 7.1, and Mw/Mn = 2.12. The polymerization activity was 81.2 Kg/mmol-Zr · hr.

Experiment C

Preparation of a catalyst solution and polymerization were performed in the manner similar to Example 1 of the present specification, except for replacing di(p-tolyl)methylene (cyclopentadienyl) (octamethyloctahydrodibenzofluorenyl) zirconium dichloride with diphenylmethylene

(cyclopentadienyl)(octamethyloctahydrodibenzofluorenyl)
zirconium dichloride.

As a result, an ethylene-1-octene copolymer was obtained with a yield of 35.2 g. The obtained polymer had a density of 902 (kg/m^3), $\text{MFR}_2 = 2.47$ (g/10 min). The polymerization activity was 70.4 Kg/mmol-Zr · hr.

Experiment D

Preparation of a catalyst solution and polymerization were performed in the manner similar to Example 1 of the present specification, except for replacing di(p-tolyl)methylene (cyclopentadienyl)(octamethyloctahydrodibenzofluorenyl) zirconium dichloride with di(p-tolyl)methylene (cyclopentadienyl) (2,7-di-tert-butylfluorenyl) zirconium dichloride.

As a result, an ethylene-1-octene copolymer was obtained with a yield of 30.6 g. The obtained polymer had a density of 902 (kg/m^3), $\text{MFR}_2 = 1.87$ (g/10 min). The polymerization activity was 61.2 Kg/mmol-Zr · hr.

5. The undersigned declares further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that

willful false statements and the like so made are punishable
by fine or imprisonment, or both, under Section 1001 of Title
18 of the United States Code and that such willful false
statements may jeopardize the validity of the application or
any patent issuing thereon.

Respectfully submitted,

this 27 day of April, 2009

Kenji SUGIMURA

Kenji Sugimura